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MEMORANDUM REPORT BRL-MR-3817

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PURIFICATION AND PROCESSING OF
ELECTROCHEMICALLY PRODUCED
HYDROXYLAMMONIUM NITRATE

NATHAN KLEIN
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MARCH 1990

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U.S. ARMY LABORATORY COMMAND

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I INTRODUCTION

The US Army is currently investigating the use of liquid propellants in large and medium caliber guns. Propellants that are aqueous and homogeneous and contain hydroxylammonium nitrate (HAN) as an oxidizer have been determined to be the most promising candidates. Propellants LGP 1845 and LGP 1846 have undergone extensive characterization and testing and consist of HAN as the oxidizer, triethanolammonium nitrate (TEAN) as the fuel, and water as a diluent and solvent. Propellant compositions are shown in Table 1.

TABLE 1

Propellant Compositicns in Weight Percent

	HAN	TEAN	H ₂ O
LGP 1845	63.2	20.0	16.8
LGP 1846	60.8	19.2	20.0

As shown in Table 1, HAN is the major ingredient in both of these propellants. There is currently only one commercial source of HAN in the United States, Southwest Analytical Chemicals, Austin, TX. The compound is sold in dilute solution (20-25 %) and must be concentrated (>80 %) for use in propellant production. Since this source produces HAN using a proprietary process not available to the US Army, the Army was compelled to develop a process for production of HAN.

II BACKGROUND

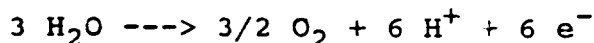
The Olin Chemicals division of the Olin Corporation was awarded a six-month contract in 1985 that was to demonstrate a feasible method for the production of high purity HAN by electrolytic reduction of nitric acid. The first task in that contract was to review the existing HAN literature in such areas as production, physical properties, toxicology, stability, environmental impact and safety. Results of this review showed that HAN had been prepared 1) in an electrodialysis cell from hydroxylammonium chloride and nitric acid; 2) from reaction of hydroxylammonium sulfate (HAS) and barium nitrate; 3) from a three-step cation exchange process employing HAS and nitric acid; 4) from the reduction of nitric acid by molecular hydrogen in the presence of a palladium catalyst; and 5) electrolytically from the reduction of nitric oxide.¹

During this feasibility study, Olin also successfully demonstrated production of HAN by the electrolytic reduction of nitric acid. The electrochemical cell was fabricated from acrylic plastic, had a mercury pool cathode, and several different anode materials. Catholyte and anolyte chambers were separated by a membrane. Temperature of the cathode chamber was controlled by a cooling chamber directly beneath the cathode. Feasibility was demonstrated and a small quantity of HAN at a concentration of about 15 weight percent was produced.

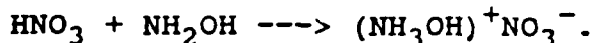
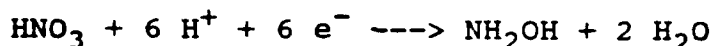
Based on the electrochemistry demonstrated during the feasibility study, Olin was awarded a contract to develop a continuous electrochemical reactor. This second, fourteen-month effort evaluated such operating parameters as anolyte and catholyte nitric acid concentrations, cathode recycle rates and catholyte temperature in order to determine their effects on cell operation

and thus optimize efficiency. A cell ten times larger than that used in the previous study was fabricated from chlorinated polyvinylchloride. As before, a horizontal mercury pool was used for the cathode and several anode materials were evaluated. The anode and cathode chambers were separated by a membrane and the cathode was cooled by a chamber beneath the cathode bedplate.

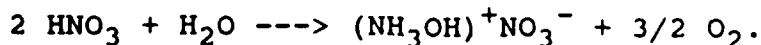
The reactions that take place in the reactor are shown below. Water in the anode chamber is oxidized to produce hydrogen ions and oxygen:



while the nitric acid in the cathode chamber is reduced to the weak base hydroxylamine. Since excess nitric acid is present, the hydroxylamine reacts to form HAN:



The overall reaction observed is thus:



Nitric acid concentrations in the two electrode chambers are critical to proper operation of the reactor. At low nitric acid concentrations hydroxylamine is reduced at the cathode to ammonia. At high nitric acid concentrations, the HAN produced is oxidized to various NO_x compounds. A balance between the nitric acid concentrations in the two electrode chambers is required in order to enhance water transport across the membrane and maximize

the efficiency of the cell. Based on these considerations, it was evident that the product leaving the cathode chamber would have to contain some excess nitric acid. The highest current efficiency was obtained in the larger scale cell when the product that exited the reactor contained approximately 25 % HAN and 2.8 % nitric acid.²

As stated earlier, the HAN concentration needed for propellant manufacture is approximately 80 % so that concentration of reactor product is required. Concentration of the 25 % HAN without removal of the excess nitric acid can result in loss of the HAN and is potentially dangerous. As water is removed from this mixture, the HAN and nitric acid react to produce NO_x . Since this reaction is exothermic, reaction rate increases as the reaction proceeds and a violent runaway reaction can readily occur. Based on these constraints, Olin set about to develop a method for removal of the excess acid. The first method attempted involved the production of free hydroxylamine from a readily available hydroxylammonium salt. Hydroxylamine vapor was to be bubbled into the reactor product. Attempts to implement this procedure in a laboratory scale apparatus were unsuccessful because of the low vapor pressure of hydroxylamine.

Sassé suggested to Olin that they try a weakly basic anion exchange resin in order to exchange hydroxide for nitrate, thus removing the excess acid.³ Olin personnel evaluated several resins and were able to generate a product with acceptable levels of nitric acid concentration. A more detailed consideration of this procedure results in cause for concern. The HAN-nitric acid mixture dissociates into hydroxylamine, hydronium, and nitrate ions. The weak base amine resin protonates and the nitrate is then absorbed on the resin leaving the free amine. HAN is obtained in the column eluant only when the ion-exchange resin is

sufficiently depleted to permit nitric acid to bleed through the resin bed and neutralize the hydroxylamine either in the collected eluant or on the column. Although the end result of this procedure is a HAN solution free of excess nitric acid, neutralization of free hydroxylamine in the resin column could pose a safety problem. The neutralization reaction is exothermic and the amine is thermally unstable. The amine decomposes to produce NO_x compounds which, in turn, can react with the resin or with other materials present on the column. If enough heat and gas is liberated in the column an explosion could result.

In considering this procedure, it would seem that the use of a strongly acidic cation exchange resin would be a more efficient approach. The use of a cation column would entail a two-stage exchange. First, the resin in its acid form would be charged, using a readily available hydroxylammonium salt such as HAS. The electrochemical reactor product would then be passed through the column, exchanging hydroxylammonium for hydronium ions. This would avoid having the neutralization reaction previously mentioned occurring in the resin bed. Sulfuric acid is the by-product of this reaction sequence.

The production of HAN through use of HAS and nitric acid in a cation exchange system is described by Wheelwright.⁴⁻⁶ The process described involves the production of HAN for use in plutonium recovery, an application in which excess nitric acid is acceptable. Although the procedure is quite similar to that needed for our specific application, it differs in that we are only using the process to purify electrolytically produced HAN rather than for manufacture of a material.

III EXPERIMENTAL METHODS

A search was undertaken for a suitable strong cation ion-exchange resin. The foremost requirement was that the resin be chemically stable to mixtures of HAN and nitric acid. Since this was to be only a feasibility study, considerations such as resin capacity, cost, and optimal operating conditions were of secondary concern. The resin IONAC CFP-110* was chosen. This material is a macroporous, sulfonated styrene-divinylbenzene copolymer and is reported by the manufacturer to be stable in moderately concentrated nitric acid. Particle size range of the resin beads is 0.35-1.2 mm.

A quantity of resin was placed in a glass chromatography column that was 4.5 cm OD and 111.8 cm high. The resin beads were fractionated by backflushing for two hours with tap water using the full column length. After settling, a bed volume of 780 mL was obtained. There was no indication of channeling in the bed. The column was prepared for use by washing with 1.5 L of 5 % nitric acid to remove any cations captured from the tap water and to insure that the resin was in its acidic form. The bed was then charged with 3 L of 5 % HAS in distilled water at a flow rate of 50 mL/min. The charged resin bed was rinsed with 2 L of distilled water at 50 mL/min and 6 L of distilled water at 200 mL/min. The column was operated at 50 mL/min with a solution of HAN and nitric acid that was 22.8 % HAN and 2.8 % acid, a mixture that closely resembles the electrochemical reactor effluent. Incremental 25 mL samples were taken and analyzed titrimetrically.⁷

* Sybron Chemicals Inc., Subsidiary of Sybron Corp.,
Birmingham, NJ.

IV RESULTS AND DISCUSSION

Neither heat evolution nor significant volume changes are observed during any operations with the resin column. When permitted to soak in 5 % nitric acid for periods of 24 hours or longer, a yellow-tan color develops in the liquid that is in contact with the beads. Although no chemical changes of the resin bed seem to be associated with this liquid color change, it would be prudent to prohibit prolonged contact of the resin with nitric acid.

The results of analysis of aliquots of the 25 mL samples taken are shown in Figure 1. The Figure presents HAN, acid, and the HAN:acid ratio, the values of the stock solution used appearing as the 0 volume values. One observes that the first 250 mL obtained are essentially pure water, this being the hold-up volume of the column. HAN then appears in the eluant, the concentration increasing to approximately 23 %. This is expected and shows that the nitric acid introduced in the stock solution is being converted to HAN on the column. After two hold-up volumes of stock solution have passed through column, some nitric acid begins to appear in the eluant indicating depletion of the resin. The resin is not fully depleted, however, until two more hold-up volumes have been eluted.

The chemical stability of the resin in contact with HAN and nitric acid was investigated by recharging the column using concentrations and flow rates described above. Since the column had been exposed to HAN and nitric acid but not to the metal ions that are present in tap water, the acid rinse that preceded charging with HAS was not included. Results obtained with the recharged column duplicate the data of Figure 1 quite precisely indicating that little or no deterioration of the resin occurred.

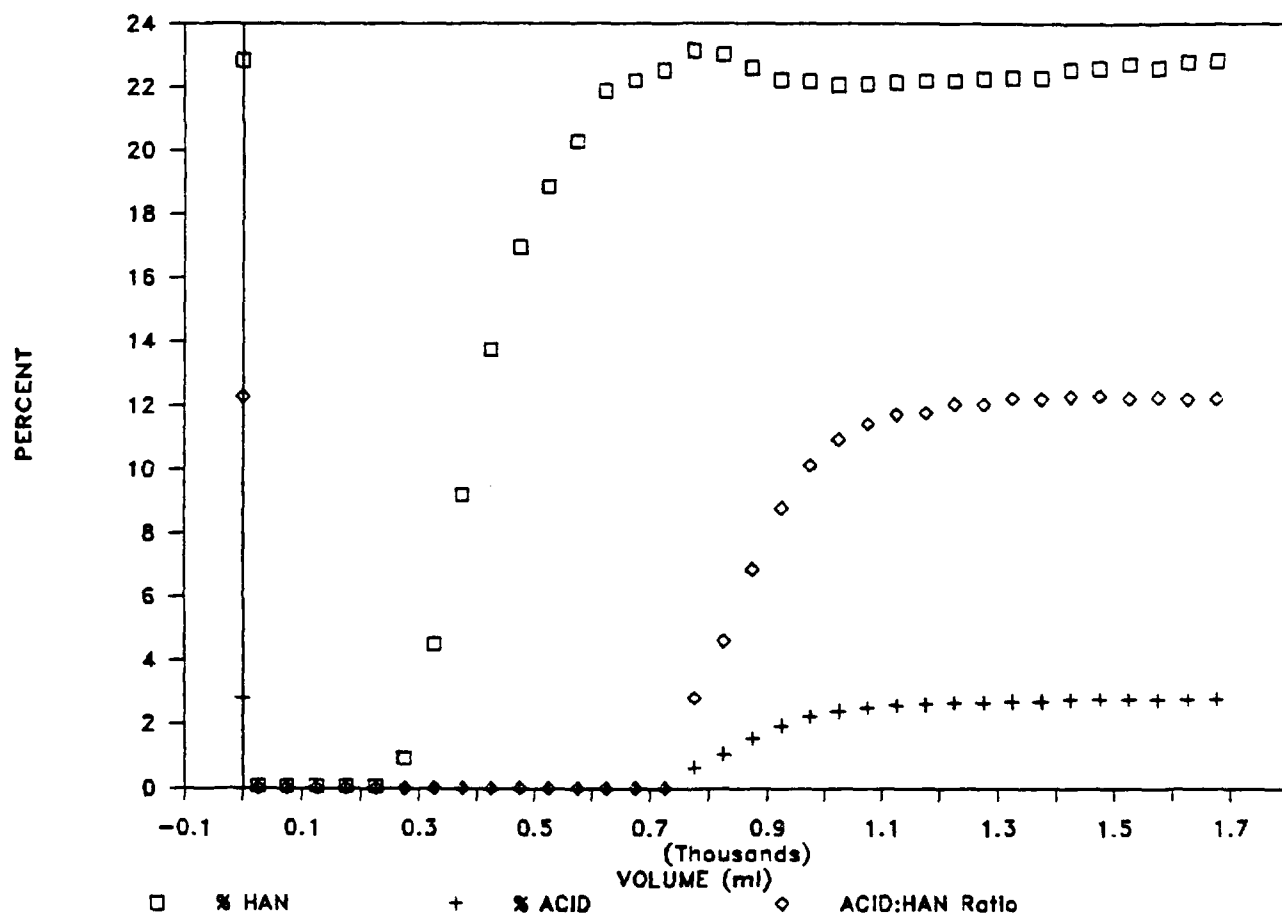


Figure 1. Performance of the CFP-110 Resin

The work described was carried out solely for the purpose of demonstrating the feasibility of an ion-exchange procedure for excess acid removal from electrolytically-produced HAN. A great deal of additional work is needed prior to its incorporation into a production facility. It would appear from the data obtained that ion-exchange columns will have to operate serially since two hold-up volumes of HAN-acid mixture are obtained before the column is totally depleted and requires recharging. Routing such partially purified eluant to a fully charged second column should increase the efficiency and reduce the overall cost of the acid removal process. In like manner, the pure water that is the first hold-up volume obtained with a freshly charged column should not be combined with the HAN solution obtained subsequently in order to avoid the expense of additional distillation. Other problems will probably surface and require attention before a practical production system for excess acid removal is completed.

Although not investigated experimentally, the use of an anion exchange resin for safe removal of excess nitric acid is also potentially feasible. The resin would be charged using aqueous ammonia. HAS would then be passed through the column producing an aqueous solution of hydroxylamine which would be used to titrate the excess nitric acid in the electrolytically produced HAN. Since the exothermic neutralization reaction does not take place on the column, the potential hazards cited earlier are avoided. Reaction by-product in this case is ammonium sulfate. The selection of cation exchange or anion exchange resins, or of a process that utilizes both cannot be recommended at this time since final selection would have to be strongly based on economic rather than technical feasibility.

V CONCLUSIONS

An ion-exchange method using a strongly acidic, cation resin for this purpose has been demonstrated. Although the resin IONAC CFP-110 was used successfully, no resin selection is implied since similar resins from other suppliers were not evaluated.

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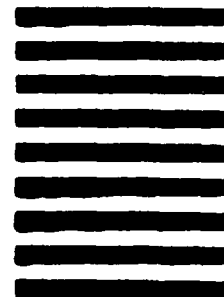
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